

FORM PTO-1390
(REV 10-95)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. §371**

TAKIT 162

U.S. APPLICATION NO. (If known, see 37 CFR §1.5)

09/937099

INTERNATIONAL APPLICATION NO.

PCT/JP00/00752

INTERNATIONAL FILING DATE

10 FEBRUARY 2000

PRIORITY DATE CLAIMED

31 MARCH 1999

TITLE OF INVENTION

ADDITIVE AND INKJET RECORDING MEDIUM USING ADDITIVES



APPLICANT(S) FOR DO/EO/US

HASEGAWA, Makoto, et al.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. §371.
 2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. §371.
 3. ☐ This express request to begin national examination procedures (35 U.S.C. §371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. §371(b) and PCT Articles 22 and 39(1).
 4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
 5. ☒ A copy of the International Application as filed (35 U.S.C. §371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
 6. ☒ A translation of the International Application into English (35 U.S.C. §371(c)(2)).
 7. ☒ Amendments to the claims of the International Application under PCT Article 34 (35 U.S.C. §371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
 8. ☐ A translation of the amendments to the claims under PCT Article 34 (35 U.S.C. §371(c)(3)).
 9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. §371(c)(4)).
 10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. §371(c)(5)).
- Items 11. to 16. below concern document(s) or information included:**
11. ☐ An Information Disclosure Statement under 37 C.F.R. §§1.97 and 1.98.
 12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 C.F.R. §§3.28 and 3.31 is included.
 13. ☒ A **FIRST** preliminary amendment.

☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
 14. ☐ A substitute specification.
 15. ☐ A change of power of attorney and/or address letter.
 16. ☐ Other items or information:

U.S. APPLICATION NO. (if known, see 37 CFR §1.55) 09/937099		INTERNATIONAL APPLICATION NO. PCT/JP00/00752		ATTORNEY'S DOCKET NUMBER TAKIT 162	
17. <input checked="" type="checkbox"/> The following fees are submitted: BASIC NATIONAL FEE (37 CFR §1.492 (a) (1) - (5)): Search Report has been prepared by the EPO or JPO..... \$860.00 International preliminary examination fee paid to USPTO (37 CFR §1.482)..... \$690.00 No international preliminary examination fee paid to USPTO (37 CFR §1.482) but international search fee paid to USPTO (37 CFR §1.445(a)(2))..... \$710.00 Neither international preliminary examination fee (37 CFR §1.482) nor international search fee (37 CFR §1.445(a)(2)) paid to USPTO..... \$1000.00 International preliminary examination fee paid to USPTO (37 CFR §1.482) and all claims satisfied provisions of PCT Article 33(2)-(4)..... \$100.00 ENTER APPROPRIATE BASIC FEE AMOUNT =				CALCULATIONS PTO USE ONLY	
Surcharge of \$130.00 for furnishing the oath or declaration later than months from the earliest claimed priority date (37 C.F.R. §1.492(e)). <input type="checkbox"/> 20 <input type="checkbox"/> 30					
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	10 - 20 =	0	x \$ 18.00	\$0.00	
Independent claims	1 - 3 =	0	x \$ 80.00	\$0.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$ 270.00		
TOTAL OF ABOVE CALCULATIONS =				\$860.00	
Reduction of 1/2 for filing by small entity, if applicable. A Verified Small Entity Statement must also be filed (Note 37 C.F.R. §§1.9, 1.27, 1.28).					
SUBTOTAL =				\$860.00	
Processing fee of \$130.00 for furnishing the English translation later than months from the earliest claimed priority date (37 C.F.R. §1.492(f)). <input type="checkbox"/> 20 <input type="checkbox"/> 30					
TOTAL NATIONAL FEE =				\$860.00	
Fee for recording the enclosed assignment (37 C.F.R. §1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 C.F.R. §§3.28, 3.31). \$40.00 per property.					
TOTAL FEES ENCLOSED =				\$860.00	
				Amount to be refunded:	
				charged:	
a. <input checked="" type="checkbox"/> A check in the amount of <u>\$860.00</u> to cover the above fees is enclosed. b. <input type="checkbox"/> Please charge my Deposit Account No. <u>13-3402</u> in the amount of \$_____ to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>13-3402</u> . A duplicate copy of this sheet is enclosed.					
NOTE: Where an appropriate time limit under 37 C.F.R. §§1.494 or 1.495 has not been met, a petition to revive (37 C.F.R. §1.137(a) or (b)) must be filed and granted to restore the application to pending status.					
SEND ALL CORRESPONDENCE TO: Customer Number 23,599					
 23599 PATENT TRADEMARK OFFICE			SIGNATURE  Anthony J. Zelano NAME		
Filed: 20 SEPTEMBER 2001			27,969		
AJZ:kmo			REGISTRATION NUMBER		

APPLICATION DATA SHEET

APPLICATION INFORMATION

Application Type:: REGULAR
Subject Matter:: UTILITY
CD-ROM or CD-R?: NONE
Title:: ADDITIVE AND INKJET RECORDING
MEDIUM USING ADDITIVES
Attorney Docket Number:: TAKIT 162

INVENTOR INFORMATION

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CORRESPONDENCE INFORMATION

Correspondence Customer Number:: 23599

REPRESENTATIVE INFORMATION

Representative Customer Number:: 23599

FOREIGN PRIORITY INFORMATION

09/937099

02 SEP 2001

Application Number::	Country::	Filing Date::	
PCT/JP00/00752	PCT	02/10/00	09970/'99
Japan	03/31/99		

ASSIGNMENT INFORMATION

Assignee Name:: Nippon Paper Industries Co., Ltd.
Street:: 4-1, Ohji 1-chome, Kita-ku
City:: Tokyo
Country:: JAPAN

09/937099

JC03 Rec'd 20 SEP 2001

(Amendment according to Article 11)

To: The President, Patent Office

- Name (Title): NIPPON PAPER INDUSTRIES CO., LTD.

Address: 4-1, Ohji 1-chome, Kita-ku, Tokyo 114-0002

Nationality: Japanese

Address: Japan

3. Agent (Representative) :

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- #### 4. Object of amendment: Specification and Claims

5. Details amendment:

(1) Line 16, page 3 of specification: " can not make to move"
is changed to "not moving".

(2) Lines 14-16 , page 5 of specification:

"1 : 0.01 - 2 : 0.5 - 2.5 : 0.00005 - 0.05 but more preferably
1 : 0.02 - 1 : 0.8 - 2.2 : 0.0001 - 0.01" is changed to

"1 : (0.01 - 2) : (0.5 - 2.5) : (0.00005 - 0.05) but more preferably 1 : (0.02 - 1) : (0.8 - 2.2) : (0.0001 - 0.01) " .

(3) The following is added to Claims 8-10.

8. The additive as defined in Claim 1, wherein the weight average

molecular weight of the cationic resin obtained is 10,000-500,000.

9. The additive as defined in Claim 1, wherein the ratio of secondary amine : ammonia: epihalohydrin : crosslinking agent is 1:(0.01-2):(0.5-2.5):(0.00005-0.05).

10. A method of manufacturing the water-resistant cationic resin, wherein a secondary amine and ammonia are first mixed in a reaction solvent, an epihalohydrin is gradually dripped in, and a crosslinking agent is then gradually dripped in.

6. Contents of appended documents

- | | |
|---------------------------|---------|
| (1) Specification, page 3 | 1 page |
| (2) Specification, page 5 | 1 page |
| (3) Claims | 2 pages |

IN THE UNITED STATES DESIGNATED/ELECTED OFFICE

International Application No. : PCT/JP00/00752
International Filing Date : 10 FEBRUARY 2000
Priority Date(s) Claimed : 31 MARCH 1999
Applicant(s) (DO/EO/US) : HASEGAWA, Makoto, et al.

Title: ADDITIVE AND INKJET RECORDING MEDIUM USING ADDITIVES

PRELIMINARY AMENDMENT

Commissioner for Patents
Washington, D.C. 20231

SIR:

Prior to calculating the national fee, and prior to examination in the National Phase of the above-identified International application, please amend as follows:

IN THE CLAIMS:

3. (Amended) An inkjet recording medium, wherein the recording medium is formed by providing an ink absorption layer on a sheet-like substrate surface which has poor ink absorption properties, and the additive according to Claim 1 is contained in said ink absorption layer.

5. (Amended) The inkjet recording medium as defined in Claim 4, wherein ordinary paper or coated paper is impregnated by a solution containing an additive having a cationic resin as principal component, this resin being obtained by the reaction of at least a secondary amine, ammonia, an epihalohydrin and a crosslinking agent.

6. (Amended) The inkjet recording medium as defined in Claim 4, wherein the ordinary paper or coated paper substrate is manufactured from a pulp slurry containing an additive having a cationic resin as principal component, this resin being obtained by the reaction of at least a secondary amine, ammonia, an epihalohydrin and a crosslinking agent.

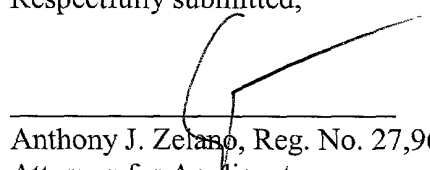
7. (Amended) The inkjet recording medium as defined in Claim 5, wherein the ordinary paper or coated paper substrate contains an additive having a cationic resin as principal component, this resin being obtained by the reaction of at least a secondary amine, ammonia, an epihalohydrin and a crosslinking agent.

REMARKS

The purpose of this Preliminary Amendment is to eliminate multiple dependent claims in order to avoid the additional fee. Applicants reserve the right to reintroduce claims to canceled combined subject matter.

Attached hereto is a marked-up version of the changes made to the claims by the current amendment. The attached pages are captioned "**Version With Markings to Show Changes Made**".

Respectfully submitted,



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AJZ:jmm

FILED: 20 SEPTEMBER 2001

VERSION WITH MARKINGS TO SHOW CHANGES MADE

Claims 3 and 5-7 have been amended as follows:

3. ~~The~~(Amended) An inkjet recording medium ~~as defined in Claim 2~~, wherein the recording medium is formed by providing an ink absorption layer on a sheet-like substrate surface which has poor ink absorption properties, and the additive according to Claim 1 is contained in said ink absorption layer.

5. (Amended) The inkjet recording medium as defined in Claim 4, wherein ordinary paper or coated paper is impregnated by a solution containing ~~the~~an additive ~~according to Claim 1~~having a cationic resin as principal component, this resin being obtained by the reaction of at least a secondary amine, ammonia, an epihalohydrin and a crosslinking agent.

6. (Amended) The inkjet recording medium as defined in Claim 4, wherein the ordinary paper or coated paper substrate is manufactured from a pulp slurry containing ~~the~~an additive ~~according to Claim 1~~having a cationic resin as principal component, this resin being obtained by the reaction of at least a secondary amine, ammonia, an epihalohydrin and a crosslinking agent.

7. (Amended) The inkjet recording medium as defined in Claim 5, wherein the ordinary paper or coated paper substrate contains ~~the~~an additive ~~according to Claim 1~~having a cationic resin as principal component, this resin being obtained by the reaction of at least a secondary amine, ammonia, an epihalohydrin and a crosslinking agent.

SPECIFICATION

Additive and inkjet recording medium using additives

Field of the Invention

5 This invention relates to an additive having a cationic resin which can render a water-soluble dye water resistant as principal component, and to an inkjet recording medium comprising this additive in the surface and/or interior of a substrate.

Background of the Invention

10 In inkjet recording, a coloured dye or coloured pigment dispersed in an aqueous or non-aqueous solvent, is extruded as minute fine drops from one or more nozzles, and the liquid drops are made to form a desired character or image by electronic control on the recording medium.

15 As a recording medium employing this recording method, paper used for electrostatic transfer or ordinary paper used for writing can be used, but to obtain a good recorded image, the recording medium must have the following two sets of characteristics. The first set of characteristics is good ink absorption, ink drops adhering to the surface of the medium rapidly penetrate the interior of the medium, the image rapidly develops a dry appearance, and the image does not smudge on contact with the recording device, another medium or the hands. 20 The second set of characteristics is that ink drops do not diffuse through the interior or surface of the medium more than is necessary, and dots recorded by the ink drops do not become

too large or assume a distorted shape.

The coloured components used in the ink may be direct dyes, acidic dyes, basic dyes, reactive dyes, dispersion dyes or various pigments. Water-soluble dyes are most often used, but
5 in the case of inkjet recording, there is usually a very serious problem insofar as the recorded image has poor water resistance. For example, when an image recorded by the inkjet recording method is taken outdoors, it may occur that of the image smudges due to rain and the recorded image can no longer be read.
10 Alternatively, if the image is left for a long period of time under high humidity conditions, it may smudge and spoil the image quality.

Hence, to make the recorded image from inkjet recording water resistant, in Tokkai Sho 55-150396 (Koho), an invention
15 is proposed wherein, after printing is performed using a water-based dye ink, a reagent is used which forms a lake with the dye to confer water resistance.

Various compounds that are ink water resistance-conferring agents and which can be first added to the ink-receiving layer of the inkjet recording medium, are also known.
20 For example,

JP,56-59239 (Koho) proposes a polycation polyelectrolyte, JP,61-68788 (Koho) proposes a weak acid salt of a polyallylamine, JP,60-49990 (Koho) proposes an ammonium
25 polyalkylene polyamine dicyandiamide, JP,1-157884 (Koho) proposes chitosan, JP,6-92011 (Koho) proposes cation denaturation colloidal silica, while JP,6-92012 (Koho) proposes a copolymer of dimethylamine and epichlorohydrin,

these compounds being added to the ink-receiving layer beforehand.

Besides the above-mentioned compounds, cationic resins such as dicyandiamide formaldehyde resin, diethylenetriamine
 5 dicyandiamide ammonium chloride condensate, (meta)acryloyloxy-alkyl trialkylammonium chloride polymer, dimethyl diallylammonium chloride polymer, ethyleneimine polymers, diallylamine polymers and ammonia/epichlorohydrin/dimethylamine copolymers, are
 10 already known as an ink water resistance-conferring agents.

It is known that the aforementioned water-soluble cationic resins form complexes by bonding to anionic water-soluble direct dyes, acidic dyes, reactive dye, etc., thereby
 15 preventing decoloring of yarn, cloth, etc. dyed with water-soluble paints.

However, if a solvent such as water is present in the environment when a complex with a dye molecule is made, the complex will displace easily from the dyed object, so these
 20 water-soluble cationic resins apparently have a low waterproofing effectiveness, and even if they were used as an ink jet recording medium, they did not give satisfactory recording quality.

It is therefore a first object of this invention to provide
 25 an additive having a cationic resin as its principal component which has a large effectiveness in improving the waterproof properties of an image colored or recorded with a water soluble dye. It is a second object of this invention to provide a

recorded medium suitable for multicolor recording by the inkjet recording method which not only addresses the problem of inadequate waterproofness of recorded characters and images, which was the defect of conventional inkjet recording media, but also improves resolution and color tone, provides a high resolution similar to that of a silver salt photograph, and gives little difference of hue of recorded characters or images compared to the hue of the dyes themselves which are currently used in ink.

Disclosure of the Invention

This invention relates to an additive having a cationic resin as principal component, the resin being obtained by reaction of at least a secondary amine, ammonia, epichlorohydrin and a crosslinking agent, and to an inkjet recording medium comprising this additive in the surface and/or interior of a substrate. The additive of this invention partly comprises a cationic resin having a crosslinked structure as principal component, hence even if a solvent such as water is present in the environment, a complex with a dye molecule cannot easily be displaced from a dyed object, and the durability of the image can be largely improved. The additive of this invention not only is effective in improving the durability of a water-based ink image, but also improves the resolution and color tone of the image. Therefore, by incorporating the additive having the cationic resin as principal component in the surface and/or interior of a substrate of a recording medium, an inkjet recording medium is obtained which not only has

excellent waterproofness and gives little blurring of dots, but also has an identical coloring hue to that of the dye itself, and allows a high resolution, high quality image to be obtained.

5 Description of the Preferred Embodiments

The cationic resin of this invention has a secondary amine, ammonia, an epihalohydrin compound and a crosslinking agent as required components, and can be obtained by their reaction. These components can be used independently, or a mixture of two or more of them may be used together.

The secondary amine used in this invention may be an aliphatic secondary amine, aromatic secondary amine or cyclic secondary amine, but an aliphatic secondary amine is to be preferred. Examples of aliphatic secondary amines are dimethylamine, diethylamine, dipropylamine, dibenzylamine, ethyl monomethylamine, methyl propylamine, butylmonomethylamine, methyl octylamine and methyl laurylamine. Of these dialkylamines, dimethylamine, diethylamine and ethyl monomethylamine are particularly desirable.

The ammonia used in this invention may be any of liquid ammonia, ammonia gas and aqueous ammonia solution, there being no particular limitation on the concentration of aqueous ammonia solution.

The epihalohydrin compound used in this invention may be epichlorohydrin, epiodohydrin or epibromohydrin, epichlorohydrin being particularly preferred.

The crosslinking agent used in this invention may be any

compound with two or more crosslinking functional groups such as aldehyde groups, epoxy groups and isocyanate groups, but compounds containing epoxy groups are to be preferred. Suitable examples are multifunctional epoxy compounds such as

5 polyethylene glycol diglycidyl ether, polypropylene glycol diglycidyl ether, polytetramethylene glycol diglycidyl ether, polybutadiene diglycidyl ether, resorcinol diglycidyl ether, neopentylglycol diglycidyl ether, 1, 6-hexanediol diglycidyl ether, bisphenol A diglycidyl ether, bisphenol F diglycidyl ether, bisphenol A polyethylene glycol diglycidyl ether,

10 bisphenol A polypropyleneglycol diglycidyl ether, hydrated bisphenol A diglycidyl ether, hydroquinone diglycidyl ether, terephthalic acid diglycidyl ether, sorbitol polyglycidyl ether, polyglycerol polyglycidyl ether,

15 pentaerythritolpolyglycidyl ether, diglyceroylpolyglycidyl ether, glycerolpolyglycidyl ether, and trimethylolpropane polyglycidyl ether.,

In the method of synthesizing the cationic resin, in JP,10-152544 (Koho), the order in which ammonia, amines and

20 epihalohydrins may be made to react is disclosed. However, in the synthesis of the cationic resin of this invention, the epihalohydrin compound is gradually dripped in after first mixing the secondary amine and ammonia together in a reaction solvent, and the crosslinking agent is then added gradually.

25 If the crosslinking agent and the secondary amine or ammonia are reacted together first, the reaction becomes uneven, and the target compound is not obtained.

In a preferred method of synthesizing the cationic resin

of this invention, the secondary amine and ammonia are dissolved under temperature less than ordinal temperature in water or an organic solvent, for example, methanol, ethanol, 2-propanol, butanol, ethylene glycol, dioxane, dimethylformamide, 2-ethoxyethanol or dimethyl sulfoxide, the
 5 epihalohydrin is dripped in, the temperature of the reaction mixture is raised to 30-100°C, the crosslinking agent is added, and the reaction is performed for 10-20 hours.

A desirable mole ratio of monomer components in the
 10 cationic resin of this invention obtained by reaction of secondary amine, ammonia, epihalohydrin compound and crosslinking agent (secondary amine : ammonia : epihalohydrin compound : cross linking agent) is 1:(0.01-2):(0.5-2.5):
 (0.00005-0.05), and preferably 1:(0.02-1):(0.8-2.2):
 15 (0.0001-0.01).

If the epihalohydrin compound is less than 0.5 mols, the secondary amine and ammonia cannot fully react with the epihalohydrin, so even if the compound obtained is used as a recording medium, it is difficult to obtain an ink jet recording
 20 target giving excellent water resistance of the recorded image. If the epihalohydrin compound is increased to exceed 2.5 mols, the resin obtained is difficultly soluble or insoluble in water, so it is unsuitable for manufacture of a recording medium in an aqueous system, and must be manufactured in a solvent system.

25 Alternatively, if the crosslinking agent is increased so that 0.05 mols are exceeded, although the reason is not clear, the desired compound cannot be obtained and it is difficult to use for manufacture of the recording medium of this invention.

Conversely, if the crosslinking agent is reduced to less than 0.00005 mols, a good inkjet recording medium having good image water resistance cannot be manufactured.

5 The average molecular weight of the cationic resin of this invention is preferably 10,000-500,000, and more preferably 50,000-250,000 as a weighted mean. The reactivity with dye molecules falls if the weight average molecular weight is larger than 500,000, and the water resistance of the record image cannot be increased. Conversely, if the weight average
10 molecular weight is less than 10,000, the reactivity with dye molecules increases too much, so when the resin comes in contact with ink, it reacts rapidly with the dye molecules in the ink to produce a precipitate, and in this case, the ink absorptivity of the recording medium falls.

15 Although the cationic resin of this invention is thought to be generated by the copolymerization of a secondary amine, ammonia, epihalohydrin compounds and a crosslinking agent, the molecular structure of this cationic resin has not yet been elucidated by the Inventor and others.

20 The state of the reaction system in the synthetic end phase of this cationic resin is solution-like or colloid-like, and the pH is 4-9. The color is light yellow, yellow, yellow brown, dark reddish-brown or brown.

The additive having the cationic resin of this invention
25 as principal component (referred to hereafter simply as additive) may be the solution obtained when the synthesis of the cationic resin composition of this invention is complete, or alternatively it may be used in the colloid state as it is.

The solvent or unreacted monomer may of course also be removed. Moreover, reagents such as a stabilizing agent or antiseptic can be added to the additive of this invention to the extent that they do not spoil the effectiveness of this invention.

5 As the substrate of the ink jet recording medium used in this invention, although paper is typical, cloth, nonwoven fabric, resin sheet, film, synthetic paper or metal sheet, etc. can be suitably chosen from the media on which ink jet recording is possible.

10 The ink jet recording medium containing the additive of this invention may be manufactured by immersing a substrate in an impregnating liquid containing the additive of this invention, then drying, or incorporating the additive of this invention in a coating liquid, applying the coating liquid to
15 the substrate and drying so as to form a layer containing the additive of this invention on the surface of the recording medium, or when the substrate is paper, by mixing the additive with pulp and other ingredients in a substrate manufacturing step in the manufacture of paper, and these methods can also
20 be combined. These methods may further be combined with known methods such as the impregnation method, coating method and internal addition method as appropriate.

25 The aforesaid impregnation liquid or coating liquid may contain further additives which are generally used, such as a filler or a binder, a pigment, a water retention agent, a water resistant agent, an optical whitening agent, pH regulator, defoaming agent, lubricant, antiseptic, surfactant or electrical conduction agent.

The drying method may be any of the usual methods, such as a steam heater, gas heater, infrared heater, electric heater, hot blast heater, microwave or cylindrical dryer. After drying, the product can be given a gloss if necessary by finishers such as a supercalender which is post processing, and a soft calender etc, which are finishing processing. In addition, it is also possible to perform general machining as may be convenient.

The ink jet recording medium obtained as described above may contain an arbitrary amount of the additive of this invention, the range of 0.1 - 25 g/m² being especially desirable. If the amount is less than 0.1 g/m², water resistance is inadequate when the medium is used in an inkjet recording device having a high ink discharge amount, and when the amount is higher than 25 g/m², the hue of the original dye and the hue of the recorded image shift so that image quality deteriorates.

The water-based ink used for ink jet recording normally comprises a water-soluble direct dye, acid dye, base dye or reactive dye as colorant together with water, lower alcohols or alkyl ethers of same as solvent. The lower alcohols are preferably polyhydric alcohols such as ethylene glycol, diethylene glycol, triethylene glycol or glycerol, and their alkyl ethers, and alkyl ethers of same are preferably low alkyl ethers such as diethylene glycol monomethylether, diethylene glycol monoethyl ether and triethylene glycol monomethyl ether. Other ink additives are for example antifungal agents, germicides, antioxidants, pH regulators, dispersants, rust preventives, chelating agents, surfactants or viscosity regulators, etc.

When the additive of this invention is used in an ink jet recording medium, anions of a dye molecule which is the coloring component in ink interacts with the additive of the invention so that a water-soluble dye becomes insoluble or difficultly soluble in water. As a result, an image recorded with high resolution as a picture or a character on the recording medium becomes water-resistant, does not smudge, and an excellent image with a small variation of hue is obtained.

Although the mechanism of the above interaction is not clear, the cationic resin obtained by copolymerization of the secondary amine, ammonia, epihalohydrin compound and crosslinking agent may form a properly developed network structure. After the reaction, this mixes with the solvent, but it is thought that after drying it becomes insoluble in water, and therefore has a far superior effect to that of the cationic resins of the prior art.

The additive of this invention can also prevent decoloring of yarn and cloth which were dyed using a water-soluble dye, and this is considered to be due to the above-mentioned reason.

Examples

Hereafter, this invention is further explained in detail by way of examples, but this invention is not limited to these. "Parts" and " %" refer to weight parts and wt % unless otherwise stated.

(Synthesis Example 1)

202.9g dimethylamine (50%), 17.6g of 24 wt% aqueous

ammonia and 310g water were introduced into a reaction vessel equipped with a stirrer, reflux condenser, dropping funnel and thermometer, and after stirring to dissolve the ingredients to obtain a homogeneous mixture, 238.6g of an epichlorohydrin compound was dripped in using the dropping funnel. After addition was complete, the mixture was reacted at 70°C for 15 hours. Next, 0.3g bisphenol A propoxylate (1 propylene oxide/phenol) diglycidyl ether was added from the dropping funnel, and reacted at 90°C for 5 hours to obtain a yellow liquefied aqueous solution containing 45% solids. The weight average molecular weight of the polymer obtained as found by high performance liquid chromatography was approximately 18,000. This will be referred to as Additive 1.

15 (Synthesis Example 2)

202.9g dimethylamine (50%), 17.6g of 24 wt% aqueous ammonia and 310g water were introduced into a reaction vessel equipped with a stirrer, reflux condenser, dropping funnel and thermometer, and after stirring to dissolve the ingredients to obtain a homogeneous mixture, 238.6g of an epichlorohydrin compound was dripped in using the dropping funnel. After addition was complete, the mixture was reacted at 70°C for 15 hours. Next, 0.2g neopentyl glycol diglycidyl ether was added from the dropping funnel, and reacted at 90°C for 5 hours to obtain a light yellow aqueous solution containing 45% solids. The weight average molecular weight of the polymer obtained as found by high performance liquid chromatography was approximately 18,000. This will be referred to as Additive 2.

(Synthesis Example 3)

202.9g dimethylamine (50%), 17.6g of 24 wt% aqueous ammonia and 310g water were introduced into a reaction vessel equipped with a stirrer, reflux condenser, dropping funnel and thermometer, and after stirring to dissolve the ingredients to obtain a homogeneous mixture, 238.6g of epichlorohydrin was dripped in using the dropping funnel. After addition was complete, the mixture was reacted at 70°C for 15 hours. Next, 0.2g 1,6-hexanediol diglycidyl ether was added from the dropping funnel, and reacted at 90°C for 5 hours to obtain a light yellow aqueous solution containing 45% solids. The weight average molecular weight of the polymer obtained as found by high performance liquid chromatography was approximately 18,000. This will be referred to as Additive 3.

(Synthesis Example 4)

202.9g dimethylamine (50%), 17.6g of 24 wt% aqueous ammonia and 310g water were introduced into a reaction vessel equipped with a stirrer, reflux condenser, dropping funnel and thermometer, and after stirring to dissolve the ingredients to obtain a homogeneous mixture, 238.6g of epichlorohydrin was dripped in using the dropping funnel. After addition was complete, the mixture was reacted at 70°C for 15 hours. Next, 0.3g hydrated bisphenol A diglycidyl ether was added from the dropping funnel, and reacted at 90°C for 5 hours to obtain a light yellow aqueous solution containing 45% solids. The weight average molecular weight of the polymer obtained as

found by high performance liquid chromatography was approximately 18,000. This will be referred to as Additive 4.

(Synthesis Example 5)

5 273.9g ethylamine (50%), 17.5g of 25 wt% aqueous ammonia
and 314.4g water were introduced into a reaction vessel
equipped with a stirrer, reflux condenser, dropping funnel and
thermometer, and after stirring to dissolve the ingredients to
10 obtain a homogeneous mixture, 238.6g of epichlorohydrin was
dripped in using the dropping funnel. After addition was
complete, the mixture was reacted at 80°C for 15 hours. Next,
1.0g bisphenol A propoxylate (1-propylene oxide/phenol)
diglycidyl ether was added from the dropping funnel, and
reacted at 90°C for 5 hours to obtain a yellow liquefied aqueous
15 solution containing 45% solids. The weight average molecular
weight of the polymer obtained as found by high performance
liquid chromatography was approximately 30,000. This will be
referred to as Additive 5.

20 (Synthesis Example 6)

150.6g dimethylamine (50%), 35.1g of 20 wt% aqueous
ammonia and 460.1g water were introduced into a reaction vessel
equipped with a stirrer, reflux condenser, dropping funnel and
thermometer, and after stirring to dissolve the ingredients to
25 obtain a homogeneous mixture, 238.6g of epichlorohydrin was
dripped in using the dropping funnel. After addition was
complete, the mixture was reacted at 90°C for 15 hours. Next,
1.0g bisphenol A propoxylate (1-propylene oxide/phenol)

diglycidyl ether was added from the dropping funnel, and reacted at 90°C for 5 hours to obtain a yellow liquefied aqueous solution containing 45% solids. The weight average molecular weight of the polymer obtained as found by high performance liquid chromatography was approximately 40,000. This will be referred to as Additive 6.

(Synthesis Example 7)

185.7g dimethylamine (50%), 35.1g of 25 wt% aqueous ammonia and 391.3g water were introduced into a reaction vessel equipped with a stirrer, reflux condenser, dropping funnel and thermometer, and after stirring to dissolve the ingredients to obtain a homogeneous mixture, 238.6g of epichlorohydrin was dripped in using the dropping funnel. After addition was complete, the mixture was reacted at 70°C for 15 hours. Next, 1.0g hydrated bisphenol A diglycidyl ether was added from the dropping funnel, and reacted at 60°C for 7 hours to obtain a yellow liquefied aqueous solution containing 40% solids. The weight average molecular weight of the polymer obtained as found by high performance liquid chromatography was approximately 50,000. This will be referred to as Additive 7.

(Synthesis Example 8)

116.1g dimethylamine (50%), 87.7g of 25 wt% aqueous ammonia and 354.3g water were introduced into a reaction vessel equipped with a stirrer, reflux condenser, dropping funnel and thermometer, and after stirring to dissolve the ingredients to obtain a homogeneous mixture, 238.6g of epichlorohydrin was

dripped in using the dropping funnel. After addition was complete, the mixture was reacted at 80°C for 15 hours. Next, 2.0g hydrated bisphenol A diglycidyl ether was added from the dropping funnel, and reacted at 80°C for 8 hours to obtain a yellow liquefied aqueous solution containing 40% solids. The weight average molecular weight of the polymer obtained as found by high performance liquid chromatography was approximately 100,000. This will be referred to as Additive 8.

(Comparative Synthesis Example 1)

202.9g dimethylamine (50%), 17.6g of 24 wt% aqueous ammonia and 310g water were introduced into a reaction vessel equipped with a stirrer, reflux condenser, dropping funnel and thermometer, and after stirring to dissolve the ingredients to obtain a homogeneous mixture, 238.6g of epichlorohydrin was dripped in using the dropping funnel. After addition was complete, the mixture was reacted at 70°C for 20 hours to obtain a yellow liquefied aqueous solution containing 45% solids. The weight average molecular weight of the polymer obtained as found by high performance liquid chromatography was approximately 20,000. This will be referred to as Additive 9.

(Comparative Synthesis Example 2)

232.2g dimethylamine (50%) and 317.4g water were introduced into a reaction vessel equipped with a stirrer, reflux condenser, dropping funnel and thermometer, and after stirring to dissolve the ingredients to obtain a homogeneous

mixture, 238.6g of epichlorohydrin was dripped in using the dropping funnel. After addition was complete, the mixture was reacted at 80°C for 15 hours to obtain a yellow liquefied aqueous solution containing 45% solids. The weight average molecular weight of the polymer obtained as found by high performance liquid chromatography was approximately 40,000. This will be referred to as Additive 10.

(Comparative Synthesis Example 3)

500.0g monoallylamine hydrochloride (60%) and 1.5g 2,2-azobis-(2-amidinopropane) dihydrochloride were introduced into a reaction vessel equipped with a stirrer, reflux condenser, dropping funnel and thermometer, and the mixture was reacted at 70°C for 15 hours to obtain a 2,2-azobis-(2-amidinopropane) crosslinked compound of a monoallylamine polymer. Next, the product was diluted with 165.2g water to obtain a yellow liquefied aqueous solution containing 45% solids. The weight average molecular weight of the polymer obtained as found by high performance liquid chromatography was approximately 18,000. This will be referred to as Additive 11.

(Comparative Synthesis Example 4)

700.0g dicyandiamide (DCDA), 600.0g diethylenetriamine (DETA), 400.0g water and 100g ammonium chloride (NH_4Cl) were introduced into a reaction vessel equipped with a stirrer, reflux condenser, dropping funnel and thermometer, and the mixture was reacted at 140°C for 4 hours. Ammonia was violently emitted during the reaction. When emission of ammonia had

subsided, heating was stopped, 600g water was added, and 270g hydrochloric acid was added to neutralize the reaction product giving a yellow liquefied aqueous solution containing 47% solids. The weight average molecular weight of the polymer
5 obtained as found by high performance liquid chromatography was approximately 10,000. This will be referred to as Additive 12.

(Application to Inkjet Recording Medium)

Manufacture of substrate of inkjet recording medium

10 12 parts of kaolin as a filler, 0.1 parts of rosin sizing agent (commercial name: Sizepin NT-76: Arakawa Chemical Co.) and 0.3 parts of aluminum sulfate, 0.3 parts of cationized starch and 0.01 parts of yield enhancing agent (commercial
15 name: Pearl Flock FR-C (Seiko Chemical Industries) were mixed with 100 parts of a pulp slurry comprising broadleaf tree bleached pulp (filtered water degree 350mlcsf) to prepare pulp slurry. Paper was manufactured from the pulp slurry obtained by a twin wire paper making machine, dried and machine calendar finished to give a raw paper of weighting 72g/m².

20

(Example 1)

Additive 1 synthesized in Synthesis Example 1 was coated on the substrate prepared as described above and dried in a size press to 0.8g/m² as dry solids, and calendar finishing was
25 performed to manufacture the inkjet recording medium of Example 1. In the coating, water was added to adjust the resin concentration so as to suitably adjust the adhesion amount on the substrate.

(Examples 2-8 and Comparative Examples 1-4)

An inkjet recording medium was manufactured exactly as described in Example 1, except that Additives 2-8 synthesized in Synthesis Examples 2-8 and Additives 9-12 manufactured in Comparative Synthesis Examples 1-4, were used instead of Additive 1 used in Example 1.

(Comparative Example 5)

10 An inkjet recording medium was manufactured exactly as described in Example 1, except that water was used for coating instead of Additive 1 used in Example 1.

(Tests)

15 Transmittance measurement

Regarding the additives synthesized in Synthesis Examples 1-8 and Comparative Synthesis Examples 1-4, the degree of mutual interaction between the ink (commercial name: Scitex 1007, Scitex black ink; Scitex 1011, Scitex red ink; Scitex 1012, Scitex blue ink; BC 121, Canon color printer ink) and the additive was evaluated by measuring the transmittance using a Shimadzu Autospectrophotometer UV3100PC (measurement wavelength: 300-800nm, slit width: 2nm, sampling interval: 0.5nm). 5 μ l of ink was dripped into 0.5ml of additive solution adjusted to 0.5% concentration, and after shaking the mixture well, it was left standing overnight. Next, it was filtered through a filter of 0.2 μ m pore size, and the transmittance of the filtrate was measured. If the substance obtained by mutual

interaction of the additive and dye is highly insoluble, it precipitates and is filtered off. It may therefore be determined that the water resistance of the recording medium containing the additive will be higher, the higher the transmittance of the filtrate. These results are shown in Table 1.

Inkjet recording suitability test

The inkjet recording media prepared in Examples 1-8 and Comparative Examples 1-5 were used to record images using a multicolor inkjet printer (commercial name: BJC-400J, Canon) and a monochrome inkjet printing system (commercial name: 6420J, SciTex Japan), and the results evaluated by the following methods are shown in Table 2.

15

(Image water resistance)

30 seconds after recording with the printer, the image was immersed in deionized water at 20°C for 30 seconds, and after allowing to stand and drying, the degree of blurring was visually evaluated. In the test results, © indicates a good result with no blurring, ○ indicates that some blurring was observed but was practically insufficient to cause a problem, △ indicates that blurring was observed and sufficient to cause a problem, and x indicates a poor result with much blurring.

25

(Resolution)

Fine lines were recorded with the printer, and visually evaluated. In the test results, © indicates a good result with

little blurring and narrow lines, ○ indicates slight blurring and slightly wider lines but practically insufficient to cause a problem, △ indicates blurring and wider lines sufficient to cause a problem, and x indicates a poor result with blurring and wide lines.

Table 1 Transmissometry result

Additive	Mole ratio	Transmittance				Note
	secondary amine /ammonia/ epichlorohydrin/ crosslinking agent	1007	1011	1012	BC21 (black)	
Synthesis Example1 (Additive1)	1:0.11:1.14:0.0002	97%	93%	92%	99%	
Synthesis Example2 (Additive2)	1:0.11:1.14:0.0003	97%	94%	95%	99%	
Synthesis Example3 (Additive3)	1:0.11:1.14:0.0003	96%	93%	95%	98%	
Synthesis Example4 (Additive4)	1:0.11:1.14:0.0003	96%	90%	94%	98%	
Synthesis Example5 (Additive5)	1:0.11:1.11:0.0007	97%	93%	94%	99%	
Synthesis Example6 (Additive6)	1:0.25:1.25:0.0008	95%	90%	94%	99%	
Synthesis Example7 (Additive7)	1:0.25:1.25:0.001	95%	90%	93%	98%	
Synthesis Example8 (Additive8)	1:1:2:0.003	92%	90%	90%	98%	
Comparative Synthesis Example1(Additive9)	1:0.11:1.14:0	90%	18%	51%	33%	
Comparative Synthesis Example2(Additive10)	1:0:1:0	95%	18%	68%	80%	
Comparative Synthesis Example3(Additive11)	—	80%	20%	40%	20%	*1
Comparative Synthesis Example4(Additive12)	—	34%	6%	3%	16%	*2

*1: 2-2-azobis-(2-amidino propane) crosslinked substance from monoallylamine polymer

*2: mole ratio: DCDA:DETA:NH₄Cl=1:0.7:0.22

Table 2 Ink jet recording aptitude evaluation

	Image durability	Resolution
Example 1	◎	◎
Example 2	◎	◎
Example 3	◎	◎
Example 4	◎	◎
Example 5	◎	◎
Example 6	○	◎
Example 7	○	◎
Example 8	○	○
Comparative Example 1	△	○
Comparative Example 2	△	○
Comparative Example 3	×	○
Comparative Example 4	×	△
Comparative Example 5	×	○

According to this invention, by incorporating an additive having a cationic resin as principal component, this resin being obtained by the reaction of a secondary amine, ammonia, an epihalohydrin and a crosslinking agent as required components, in the surface and/or interior of a substrate of a recording medium, an inkjet recorded image which has water resistance, little blurring and high resolution, can be obtained.

Industrial Field of Application

By using the additive of this invention, a recording medium suitable for multicolour recording by the inkjet recording method, giving little shift in hue, high resolution

and excellent image reproducibility including color reproducibility can be obtained. Further, the additive of this invention prevents fading of dyed yarn or cloth when water-soluble dyes are used.

What is Claimed Is:

1. An additive having a cationic resin as principal component, this resin being obtained by the reaction of at least a secondary amine, ammonia, an epihalohydrin and a crosslinking agent.

2. An inkjet recording medium, comprising the additive as defined in Claim 1 in the surface and/or interior of a sheet substrate.

3. The inkjet recording medium as defined in Claim 2, wherein the recording medium is formed by providing an ink absorption layer on a sheet-like substrate surface which has poor ink absorption properties, and the additive according to Claim 1 is contained in said ink absorption layer.

4. The inkjet recording medium as defined in Claim 2, wherein the sheet-like substrate is ordinary paper, or a coated paper comprising a recording layer suitable for recording by a water-based ink on the substrate surface.

5. The inkjet recording medium as defined in Claim 4, wherein ordinary paper or coated paper is impregnated by a solution containing the additive according to Claim 1.

6. The inkjet recording medium as defined in Claim 4, wherein the ordinary paper or coated paper substrate is manufactured from a pulp slurry containing the additive according to Claim

1.

7. The inkjet recording medium as defined in Claim 5, wherein
the ordinary paper or coated paper substrate contains the
5 additive according to Claim 1.

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY
(Includes Reference to PCT International Applications)

ATTORNEY'S DOCKET NUMBER

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought of the invention entitled:

ADDITIVE AND INK-JET RECORDING MEDIUM CONTAINING THE SAME

the specification of which (check only one item below):

☒ is attached hereto.

☐ was filed as United States application

Serial No. _____

on _____

and was amended

on _____ (if applicable).

☐ was filed as PCT international application

Number PCT/JP00/00752

on February 10, 2000

and was amended under PCT Article 19

on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim priority benefits under Title 35, United States Code, §119 of the following United States Provisional Application and of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR U.S. PROVISIONAL AND FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. 119:

COUNTRY (if PCT indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 USC 119
JAPAN	09970/99	Marh 31, 1999	<input checked="" type="checkbox"/> YES <input type="checkbox"/> NO
			<input type="checkbox"/> YES <input type="checkbox"/> NO
			<input type="checkbox"/> YES <input type="checkbox"/> NO
			<input type="checkbox"/> YES <input type="checkbox"/> NO
			<input type="checkbox"/> YES <input type="checkbox"/> NO

Combined Declaration For Patent Application and Power of Attorney (Continued)
(Includes Reference to PCT International Applications)

ATTORNEY'S DOCKET NUMBER

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

U.S. APPLICATION NUMBER	U.S. FILING DATE	PATENTED	PENDING	ABANDONED

PCT APPLICATION NO.	PCT FILING DATE	U.S. SERIAL NUMBERS ASSIGNED (if any)			

POWER OF ATTORNEY: As a named inventor, I hereby appoint I. William Millen (19,544); John L. White (17,746); Anthony J. Zelano (27,969); Alan E.J. Branigan (20,565); John R. Moses (24,983); Harry B. Shubin (32,004); Brion P. Heaney (32,542); Richard J. Traverso (30,595); John A. Sopp (33,103); Richard M. Lebovitz (37,067); John H. Thomas (33,460); Catherine M. Joyce (40,668); James T. Moore (35,619); James E. Ruland (37,432); Nancy Axelrod (44,014) and Jennifer J. Branigan (40,921) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

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Combined Declaration for Patent Application and Power of Attorney (Continued) <small>(Includes Reference to PCT International Applications)</small>				ATTORNEY'S DOCKET NUMBER
20	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
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	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY
21	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY
22	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY
23	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY
24	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY
25	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
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<p>I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.</p>				
SIGNATURE OF INVENTOR 201		DATE	SIGNATURE OF INVENTOR 207	
		Sep-05-01		
SIGNATURE OF INVENTOR 202		DATE	SIGNATURE OF INVENTOR 208	
		Sep-05-01		
SIGNATURE OF INVENTOR 203		DATE	SIGNATURE OF INVENTOR 209	
SIGNATURE OF INVENTOR 204		DATE	SIGNATURE OF INVENTOR 210	
SIGNATURE OF INVENTOR 205		DATE	SIGNATURE OF INVENTOR 211	
SIGNATURE OF INVENTOR 206		DATE	SIGNATURE OF INVENTOR 212	